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TITLE: METHOD FOR PURIFYING NATURAL CRUDE WAX

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ABSTRACT:
PURPOSE: To effectively remove unnecessary ingredients from a natural crude wax by adding a given amount of a solvent to the wax, heating the mixture to around the melting point of the wax to dissolve the wax, cooling the solution to precipitate a solid matter, and conducting solid-liquid separation so that the wax recovered has a given solvent content.

CONSTITUTION: A solvent (e.g. a 1-4C alcohol) is added to a crude wax (e.g. one obtained from sugar cane) in an amount of 3-30 times by weight. The mixture is heated to a temp. in the range of [melting point of the wax] ±20°C to dissolve the wax. The solution is cooled to -10°C to 40°C to precipitate a solid matter. The solid is separated from the liquid with a centrifugal separator, etc., so that the solvent content of the solid becomes 50-90%. A solvent is added to solid thus obtained in an amount of 3-20 times by weight, and the mixture is heated to a temp. in the range of [melting point of the wax] ±20°C to dissolve the wax. The resulting solvent-insoluble matter containing an insoluble high-density substance is removed by solid-liquid separation at such a temp. The solvent is removed from the resulting wax solution by distillation, etc.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] this invention relates to the refining technique of a natural rough wax. Furthermore, a natural rough wax is refined in detail using a solvent, and it is related with the refining technique of the natural rough wax for considering as the wax material for which it was suitable in the intended-use field of various waxes.
[0002]
[Description of the Prior Art] Remove the pitch which a natural rough wax contains by melting, using a solvent as the refining technique of a natural rough wax, and filtering this further conventionally, or (JP,2-279794,A), Cool, once it melts a rough wax in the organic solvent, and a part for a wax is made to separate. After only filtering this, there was a method (JP,2-150496,A) of distilling a part for the wax which ****ed and collecting solvents etc., and it has been applied to carnauba wax, a candelilla wax, etc. which are mainly characteristic also of a natural wax.
[0003]
[Problem(s) to be Solved by the Invention] However, like the above-mentioned conventional method, by the technique by the simple filtration process, since it was difficult, fully removing the impurity which various natural rough waxes contain could not but repeat the same refining process twice [at least] or more. However, it is difficult to decrease the content of the unnecessary component which is simply soluble in a solvent and remains only by performing precipitation operation and filtration operation since the property of a natural rough wax changes with the composition remarkably below to a constant value. Therefore, by the above technique, a homogeneous wax is not obtained but the problem was in the quality of recovery or a product.
[0004]
[Means for Solving the Problem] The result to which this invention persons repeated the study zealously in view of such a situation about specialization and those effective elimination technique of the detrimental component which makes the property of a refining wax unstable in the refining processing process of a natural rough wax, The resin-like component and glyceride which contain an unnecessary component in a natural rough wax, It finds out that it is a part for a part for a part for the unsaturated fatty acid of disengagement, and the sterol of disengagement, and the sterol ester which these fatty acids and the sterol combined. After performing and the following process (I), it found out that the above-mentioned unnecessary component could be effectively removed by performing a process (II), and the technique of manufacturing a homogeneous natural wax industrially is established, and it came to complete this invention.
Namely, the 1st of this invention, It is related with the refining technique of the natural rough wax characterized by consisting of the following process (I).
Process (I): The process which separates and collects these solid contents by solid-liquid-separation operation so that the solvent content in the wax solid content which added the twice [3 - 30 weight] as many solvent as this to the natural rough wax containing an insoluble high density material, melted a part for a wax by warming to the temperature requirement of 20 degrees C of the upper and lower sides of the melting point of this wax, was made to separate a wax solid content by subsequently cooling, and were collected may become 50 - 90%. Moreover, the 2nd of this invention related with the refining technique of the natural rough wax characterized by consisting of the following process (I), a process (II), and a process (III).
Process (I): A twice [3 - 30 weight] as many solvent as this is added to the natural rough wax containing an insoluble high density material. A part for a wax is melted by warming by the temperature requirement of 20 degrees C of the upper and lower sides of the melting point of this wax. Subsequently, the process which separates and collects these solid contents by solid-liquid-separation operation so that the solvent content in the wax solid content which was made to separate a wax solid content and were collected by cooling may become 50 - 90%, Process (II): A twice [3 - 20 weight] as many solvent as this is added to the wax solid content obtained at the process (I). A part for a wax is melted by warming to the temperature requirement of 20 degrees C of the upper and lower sides of the melting point of a wax.

The process which separates and removes a part for the solvent insoluble solution which contains an insoluble high density material from a solution by solid-liquid-separation operation while holding to this temperature requirement, and the process which separates and collects refining waxes by removing a solvent from the wax solution which obtained by process (III):process (II).

[0005] this invention is explained further below. The unnecessary component in an introduction natural rough wax is explained. These consist of a part for a part for a part for the unsaturated fatty acid of a resin-like component, glyceride, and disengagement, and the sterol of disengagement, and the sterol ester which these fatty acids and the sterol combined as mentioned above, bar crystallization of a wax, and spoil hardness. Moreover, what has the low melting point among the above-mentioned matter becomes the cause of reducing the melting point of the whole wax. As for an unstable component, the structure, such as a thing containing a unsaturated bond, itself causes the instability of a wax heat, an oxidizing atmosphere, etc. Regardless of a vegetable system and an animal system, if the refining technique of the natural rough wax of this invention is a natural rough wax, it is applicable to all. For example, it produces to nature, such as animal system waxes, such as vegetable system wax; spermaceti wax, such as a cane wax, a bead wax, a rice wax, carnauba wax, and a candelilla wax, or a sheep wool low, and a rough wax to be refined is illustrated. The wax with which the technique of this invention is adopted suitably especially is a vegetable system wax, and is a rough wax from a cane still suitably.

[0006] Here, the typical process of a cane rough wax is shown. Gentle placement of the sugar sap which squeezed and squeezed out the cane in the raw sugar manufacturing process surfaces the suspended matter which mainly becomes the sap upper part from a part for a part for fiber, and fats and oils. The filter cake which carries out the filtration separation of this and is obtained is dried, and a xeransis cake is obtained. It extracts to this xeransis cake, using the light organic solvent, for example, a hydrocarbon, as an extracting solvent. After completing an extraction, it filters immediately and the separation elimination of the filtration residue is carried out. Filtrate is put, the water layer of a lower layer is separated, a solvent layer is distilled, and if a solvent is removed, a cane rough wax will be obtained. Furthermore, the stalk front face of a cane is scratched and it can also consider as the object of refining of the cane rough wax extracted and obtained by a warm temperature water bath or the organic solvent. In addition, if it is the natural rough wax which needs refining, the purification method of this invention is applicable also to which thing.

[0007] In the process (I) of the refining technique of the natural rough wax of a <process (I)> this invention A part for the unsaturated fatty acid of glyceride and disengagement which is an unnecessary component contained in a natural rough wax, The property to be hard to crystallize in case the amount of [which a part for a sterol, and these fatty acids and sterol of disengagement combined] sterol ester etc. is the recrystallization of a wax is used. By fully carrying out the solid liquid separation of these unnecessary components in the status [having melted into a solvent], the unnecessary component in a wax can be removed effectively and the wax which was very excellent as a result can be obtained.

[0008] Furthermore, it is as one of the characteristic features of this invention. Performing a process (I) in advance of a process (II) is mentioned. As mentioned above, specific gravity is larger than a wax solution so that the resin-like component may be contained to some extent in the natural rough wax and it may understand from sedimenting automatically if it melts in a solvent and this component puts a wax. Then, sedimentation of the wax solid content mixed for the wax [a resin-like component] is formed by cooling, agitating this slowly as it is, after changing into the status that it has suspended, without adding a solvent to a natural rough wax, melting a part for a wax in it uniformly, and a resin-like component being soluble in it, and making a part for a wax separate. This wax solid content becomes larger [since a resin-like component is included, a density is large, and] than the case where the density difference with a solution does not contain a resin-like component. Moreover, it becomes the nucleus at the time of a resin-like component being wax crystallization, and the large crystal of grain is obtained. Thus, by carrying out the solid liquid separation of the slurry liquid which consists of a solution and a wax solid content, it is effectively separable into a solution and a wax solid content. As the technique of a separation, the sedimentation using the density difference, especially the centrifugation separation are effective, and by including a resin-like component, since the crystal grain child is firm, a filtration separation, especially a vacuum filtration, a centrifugal-filtration separation, etc. are applicable. Thus, in the solution separated with the wax solid content, the unnecessary component which spoils the hardness of a wax is contained alternatively, and the performance of the wax solid content which carried out separation recovery as a hard wax improves.

[0009] However, when it is required for the content of the above-mentioned resin-like component to perform still highly to be too few **** or a refining separation, in advance of the process (II), it is only inadequate in the process (I) just to carry out. Therefore, it sets at the process (I) of this invention. An insoluble high density material is added to a solvent. This insoluble high density material demonstrates the same effect as the above-mentioned resin-like

component. That is, this high density material serves as the nucleus in the case of wax crystallization, and forms the large crystal of grain. Moreover, by carrying out the solid liquid separation of the slurry liquid which consists of a solution and a wax solid content, it is effectively separable into a solution and a wax solid content. Therefore, effective refining will be attained if an insoluble high density material is added to a solvent by the technique of this invention even if even if it is in the case with few contents of the resin-like component in a rough wax.

[0010] The insoluble high density material added here requires that to be the thing to which the density of a wax solid content is made to increase should be demanded, and it should have a density more than a wax and an EQC at least. If it does not melt in the solvent to use substantially and a wax also has a density and a density more than an EQC, such an insoluble high density material can use anything, and will not be limited especially. Moreover, not only the role of a mere coagulant or a flocculant but the work as a habit modifier has this matter.

[0011] Usually, a mineral matter can be used as an insoluble high density material, for example, salts, such as a carbonate and a sulfate, are further illustrated for the oxide of metals, such as aluminum, silicon, magnesium, calcium, barium, titanium, and a zirconium, a chloride, a sulfide, etc. A thing with a particle size of 0.1 micrometers - 10mm is used for all, and the addition is 0.1 - 50% of the weight of a domain. The above-mentioned insoluble high density material may have the function to adsorb the tinction component in a wax. There is a porous metallic oxide etc. as an example of such matter, and a well-known metallic oxide etc. is conventionally mentioned as adsorbents, such as aluminas, such as a zeolite which is specifically a crystalline aluminosilicate, and a titania, a zirconia, and a magnesia. Moreover, although an adsorbent, active carbon, etc. of an organic system can also be used if needed, processing of adding an additive further is needed so that these matter may have a density larger than a wax solution. Furthermore, you may have simulataneously the function which neutralizes the acid in a wax like alkaline solids, such as slaked lime. The above insoluble high density material, In a process (I), a wax solid is formed stably at least, and it may dissociate from a wax as a part for a solvent insoluble solution in a process (II).

[0012] It sets at the process (I) of the refining technique of the natural rough wax of this invention. After adding and warming a twice [3 - 30 weight] as many solvent as this in a raw material wax and melting a part for a wax in it, this is cooled and it is required to carry out the solid liquid separation of the wax solid content made to separate so that the solvent content in a wax solid content may become 50 - 90%. the amount of the solvent which melts a wax -- the weight of a raw material wax -- receiving -- the amount of three to 30 times -- an amount is used five to 20 times more preferably To a wax, the solid liquid separation of the wax and solvent which were separated by cooling cannot become difficult as a matter of fact, and the amount of a solvent cannot separate an unnecessary component, when fewer than 3 weight twice. Moreover, although the elimination factor of an unnecessary component improves so that many solvents are used, since the elution volume of a wax component also increases simultaneously and the loss for a wax increases, a solvent-recovery cost is also taken into consideration and 30 weight twice are a limitation to a wax practical. In addition, when the wax to supply contains a solvent, the amount of solvents which also takes into consideration and adds the amount of solvents to contain is determined. Therefore, when the amounts of solvents run short, it adds separately, and in being superfluous, proper technique, for example, distillation etc., adjusts in the amount of solvents of the optimum dose, and it uses it.

[0013] In the process (I), although near ordinary temperature is used, as the temperature at the time of making a wax solid content separate by cooling is low, it is more advantageous to the precipitation of a wax. However, since an unnecessary component will separate and selectivity will fall if temperature is too low not much although based also on the solubility of the solvent to a wax, -10 degrees C or more of the temperature of 0 degrees C or more are usually used preferably. Moreover, if the temperature in the case of cooling is too high, a precipitation of a wax solid content is not enough, and since a solvent becomes is easy to be included into the separated wax solid content, cooling temperature must be 40 degrees C or less.

[0014] After melting a raw material wax in a solvent in the above-mentioned process (I) A solid liquid separation is performed so that it may cool and the solvent content in this solid content may become 50 - 90% about the wax solid content made to separate., When there are more solvent contents in the wax solid content after a solid liquid separation than 90%, the unnecessary component which makes the property of a wax unstable is not fully removed, but the quality of a product wax deteriorates. Moreover, although it is more desirable as there are few amounts of solvent remains in a wax, 50% is a limitation when practical use conditions, such as equipment, are taken into consideration. It is not desirable from the purpose which a solvent is removed more than it, and the time and the cost for a way, then refining increase, and obtains a uniform quality. A more desirable result will be obtained if solid-liquid-separation operation is performed so that the solvent content in a wax solid content may become 60 - 80% of a domain practically. Therefore, it sets at the process (I) of the manufacture technique of the natural rough wax of this invention. Solid-liquid-separation operation is performed so that the solvent content in this solid content may become 60 - 80% of a domain preferably 50

to 90% about the wax solid content which made the raw material wax cool and separate this after warming in a twice [5 - 20 weight] as many solvent as this more preferably and melting a part for a wax, 3 - 30 weight twice and.

[0015] It sets at the process (I) of this invention. Although the suction filtration used, for example by usual filtration operation as technique of performing a solid liquid separation can also be used so that the solvent content in this solid content may become 50 - 90% about the wax solid content made to cool and separate after melting a raw material wax in a solvent, the method of performing a solid liquid separation more compulsorily from requiring most time for reaching the above-mentioned solvent content is desirable. It is desirable to apply the equipment which specifically applies and filters pressures, such as the equipment which separates a solid content and a liquid compulsorily using centrifugal forces, such as centrifugal-filtration equipment and a centrifugal separator, or the filter press, and a belt press, a screw press.

[0016] by the way the wax obtained from a process (I) -- since a high density material insoluble to a part of resin-like components and solvents added separately exists in a wax solid content if it remains as it is, it is necessary to remove these further and, for this reason, the following process (II) is performed However, it may not become failure especially even if an insoluble high density material etc. exists in the solvent of the resin-like component which remains in part, or the above added separately by some intended use of a refining wax. When such, it is not necessary to use a process (II). It corrects. Since the solvent content in a wax is 50 - 90%, the wax obtained from a process (I) can be used as a refining wax by removing a solvent by conventional methods, such as distillation.

[0017] The process (II) of the refining technique of the natural rough wax of a <process (II)> this invention A part for the solvent insoluble solution which melts a wax in a solvent uniformly in near the melting point of a wax, and is contained in the wax to this, The resin-like component contained in the insoluble high density material added by (I), i.e., a process, or a rough wax can use the property to be hard to melt in a solvent, and, thereby, can remove effectively a part for the solvent insoluble solution in a wax. That is, in the process (II) of this invention, a twice [3 - 20 weight] as many solvent as this is added to a raw material wax, and solid-liquid-separation operation removes a part for a solvent insoluble solution, melting a part for a wax and holding to this temperature by warming to the temperature within the limits of 20 degrees C of the upper and lower sides of the melting point of a wax. In order to remove effectively a part for the solvent insoluble solution in a wax, it is important to make the amount of a solvent one 3 to 20 times the amount of this to the weight of a raw material wax. Even if a solvent cannot separate a part for an insoluble solution effectively less than [3 weight twice] and it exceeds and adds 20 weight twice, a remarkable effect is not demonstrated but becomes disadvantageous economically. A desirable result is obtained by using a twice [5 - 10 weight] as many solvent as this for a raw material wax, and removing a part for an insoluble solution practically. When the wax to supply already contains a solvent, the amount of solvents which also takes into consideration and adds the solvent to contain is determined. That is, when the amounts of solvents run short, it adds separately, and in being superfluous, beforehand, proper technique, for example, distillation etc., adjusts in the amount of solvents of the optimum dose, and it uses it.

[0018] In the above-mentioned process (II), in case a rough wax is melted in a solvent, temperature is held in the domain of 20 degrees C of the upper and lower sides of the melting point of a wax. As long as it melts by the above-mentioned temperature requirement, it can melt in a solvent uniformly and the amount of wax can separate the solid content of non-solubility into a solvent from a solution in the stable status. Since 20 degrees C is exceeded, and the temperature to melt also separates a part for a wax from the melting point of a wax in being low, a separation with a part for a solvent insoluble solution becomes difficult. Moreover, if 20 degrees C is exceeded and it melts at high temperature from the melting point, the result which adds too much heat history to the wax which a possibility that an unnecessary component may be eluted out of the above-mentioned solvent insoluble solution part is upwards, and is melted is brought, and it is not desirable.

[0019] In the above-mentioned process (II), the well-known solid-liquid-separation technique can be conventionally used as technique of removing a part for a solvent insoluble solution, holding a raw material wax in the domain of 20 degrees C of the upper and lower sides of the melting point of a wax. Specifically, although usual filtration operation, sedimentation, etc. can also be used, in order to fully carry out, quick and the method of performing a solid liquid separation compulsorily, for example, a vacuum filtration, centrifugal separation, a centrifugal-filtration method, etc. are more desirable in operation.

[0020] If the solvent used in the process (I) of the refining technique of the natural rough wax of this invention and (II) is a solvent generally used for solvent refining of waxes, all can be preferably used for it. Especially the lower alcohol is excellent in fields, such as selectivity, and the alcohol of 1 to 4 has a desirable carbon number especially. As an example of these alcohols, a methanol, ethanol, 1-propanol, 2-propanol, 1-butanol, 2-butanol, tert-butanol, etc. are mentioned. In the above-mentioned process (I) and (II), a solvent different, respectively may be used and the same

solvent may be used at both processes.

[0021] It sets to the refining technique of the natural rough wax of this invention. A process (II) is performed after performing the above-mentioned process (I). Furthermore, according to the quality required of the property and refined material of a raw material wax, it can be suitably used for a process (I), the interval of (II), or order combining other refining technique, for example, a hydrorefining, adsorption operation, etc. A process (II) is performed after performing a process (I), The following effects are acquired. That is, since the density difference of a solution and a wax solid content increases and a solid liquid separation becomes easy by incorporating intentionally the resin-like component which exists in a natural rough wax in a process (I) in a wax solid content, or adding an insoluble high density material and incorporating in a wax solid content, the amount of the solvent to use can be lessened. Since the above-mentioned effect is not acquired on the other hand when performing a process (I) after a process (II) The density of a solution is lowered in a process (I), in order to extend the density difference with a wax solid content and to improve a separative power, a solvent must be used in large quantities, and it becomes disadvantageous economically.

[0022] Since the wax solution melted in the solvent is obtained from the <process (III)> aforementioned process (II), in a process (III), a solvent is separated and removed, for example by conventional methods, such as a precipitation by distillation or cooling. A refining wax is manufactured by passing through this process.

[0023]

[Example] this invention is not limited by these although an example explains this invention in detail below.

<Example of a reference manufacture> The sugar sap which squeezed and squeezed out the cane by the extraction raw sugar manufacturing process of a cane rough wax was put, the filter cake which carries out the filtration separation of the suspended matter which surfaces in the sap upper part, and which mainly consists of a part for a part for fiber and fats and oils, and is obtained was dried by the vacuum thermostat, and the xeransis cake was obtained. This xeransis cake was put into the container equipped with the agitator, the reflux condenser, and the heating heater, and it heated, adding and agitating n-heptane as an extracting solvent, and extracted at the reflux temperature of a solvent. After completing an extraction, it filtered immediately and the separation elimination of the filter cake was carried out. After having put filtrate and separating the water layer of a lower layer, the heptane layer was distilled, the solvent was removed and the cane rough wax of a dark green color was obtained. The peaks main 67.0 degree C in the result of DSC (differential scanning calorimetric analysis) of the above-mentioned cane rough wax were seen, and also a peak is accepted in 49 degrees C and no less than 76 degrees C, and it turns out that a melting-temperature domain is wide with broadcloth as whole. Moreover, gas-chromatograph analysis showed that the component unnecessary as a wax was contained about 40%.

[0024] The cane rough wax obtained in the above-mentioned example of a reference manufacture was put into the container equipped with the <example 1> process (I):reflux condenser, the agitator, and the heating heater, and ********** addition of the calcium hydroxide for the acid-number equivalent of a rough wax was carried out at this. It agitated for 1 hour, continuing ****** and warming for ethanol 5 times to a wax weight, and making a solvent flow back as a solvent, after having warmed the container, having melted the wax and making it react with a calcium hydroxide, and the wax was melted further. It was left for 24 hours, having cooled, agitating slowly, having lowered temperature to 5 degrees C, and agitating slowly at a room temperature further, after checking that the wax had melted into the solvent. The solid liquid separation was performed for the slurry-like liquid which the wax separated for 10 minutes on condition that 1500G using the centrifugal filter. At this time, the amount of solvent remains in a wax solid content was 72%. The solvent was removed from the obtained wax solid content by distillation, and the refining cane wax (S) was obtained.

Process (II): The refining cane wax (S) obtained at the above-mentioned process (I) was put into the container equipped with the reflux condenser, the agitator, and the heating heater, and it agitated for 1 hour, 5-weights-******ing, and a wax warming 2-propanol as a solvent, and making a solvent flow back, and the wax was melted. At this time, the temperature in a solution was temperature higher about 4 degrees C than the melting point of a wax. The centrifugal separator performed the separation for this for 10 minutes on condition that 2000G, keeping temperature at 65-70 degrees C, after checking that the wax had melted into the solvent. The solid content and wax solution containing the calcium-hydroxide reactant which sedimented were isolated preparatively by the decantation, the solvent was removed by distillation, and the refining cane wax (T) was obtained. A sharp peak single 78.7 degree C in the result of DSC of a refining cane wax (T) is seen, and it turns out that it is a wax with very sharp synchysis nature. Moreover, gas-chromatograph analysis showed that a component unnecessary as a wax was 5% or less. Furthermore, the acid number of a wax was 7.0 (mg-KOH/g-wax), and was the character which was excellent as a natural wax refined material of the low acid number.

[0025] The cane rough wax obtained in the aforementioned example of a reference manufacture was put into the

container equipped with the <example 2> process (I):reflux condenser, the agitator, and the heating heater, and ********** addition of 10% of the weight of the aluminum oxide of a rough wax was carried out at this. A container is warmed and a wax is melted. It agitated for 1 hour, continuing ****** and warming for 2-propanol 5 times to a wax weight, and making a solvent flow back as a solvent, after mixing with an aluminum oxide, drawing in to about 20 mmHgs with a vacuum pump, and the wax was melted further. It was left for 24 hours, having cooled, agitating slowly, having lowered temperature to 5 degrees C, and agitating slowly in a room temperature further, after checking that the wax had melted into the solvent. The solid liquid separation was performed for the slurry-like liquid which the wax separated for 10 minutes on condition that 2000G using the centrifugal separator. At this time, the amount of solvent remains in a wax solid content was 71%. The solvent was removed from the obtained wax solid content by distillation, and the refining cane wax (U) was obtained.

Process (II): It refining cane wax [which was obtained at the above-mentioned process (I)] (U) Put into the container equipped with the reflux condenser, the agitator, and the heating heater, and it agitated for 1 hour, 5-weights-******ing, and a wax warming 2-propanol as a solvent, and making a solvent flow back, and the wax was melted. At this time, the temperature in a solution was temperature higher about 5 degrees C than the melting point of a wax. The centrifugal separator performed the separation for this for 10 minutes on condition that 2000G, keeping temperature at 65-70 degrees C, after checking that the wax had melted into the solvent. The solid content and wax solution containing the aluminum oxide which sedimented were isolated preparatively by the decantation, the solvent was removed by distillation, and the refining cane wax (V) was obtained. A sharp peak single 79.3 degree C in the result of DSC of a refining cane wax (V) is seen, and it turns out that it is a wax with very sharp synchysis nature. Moreover, gas-chromatograph analysis showed that a component unnecessary as a wax was 5% or less. Furthermore, the hue of a wax was yellow and was a hue which was excellent as a natural wax.

[0026]

[Effect of the Invention] The process (I) of this invention is performed. By using refining operation of subsequently performing a process (II), the unnecessary component in a natural rough wax can be removed effectively, and the wax which was very excellent can be obtained.

[Translation done.]